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TOPICAL REPORT ACCO-42

THE DEVELOPMENT OF A RESIN-IN-PULP PROCESS AND ITS APPLICATIONS TO ORES OF THE WHITE CANYON AREA OF UTAH

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ABSTRACT

This is a description of a process and of the development of the process and of a machine for the recovery of uranium from acid slurries without accomplishing a liquid-solid separation. Metallurgical results obtained from testing two ores from the White Canyon Area of Utah with this process are given. The two ores tested were, White Canyon Copper Uranium ore and ore from the Happy Jack Mine.

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I. INTRODUCTION

Soon after the adsorption of uranium by anion exchange resins was discovered, it was recognized that the direct adsorption of uranium from ore pulps was mechanically and metallurgically possible. Successful development of resin-in-pulp treatment would eliminate thickening or filtration. Early bench scale tests by the Massachusetts Institute of Technology under Professor A. M. Gaudin[1/] and later work by the American Cyanamid Company,[2/] indicated that adsorption was no different in ore pulps than in clear solutions. The discovery that elution could be effected by either batch operation or by continuous flow of elutriant through beads under mild agitation demonstrated that the process was chemically sound.

In view of these promising development, a pilot plant was built in a small building outside of the Watertown Laboratory of the American Cyanamid Company in early 1952. Results of test runs on ores from South Africa and the Colorado Plateau were most encouraging.[3/] It was demonstrated that uranium could be effectively removed from an acid ore pulp during its passage through a series of machines in which exchange resin was retained by screens in moving baskets. Because of space limitations and the resultant difficulty in leaching sufficient ore to test the process thoroughly, it was decided that a much larger plant should be built somewhere close to a source of ore.

On July 18, 1952, in a conference in Washington, D. C., Mr. D. M. Kentro of the American Cyanamid Company proposed to Mr. J. C. Johnson, Director of the Raw Materials Division, U. S. Atomic Energy Commission, and Dr. George G. Marvin, Assistant Director for Process Development, that a five ton per day pilot plant be built on the A.E.C. compound in Grand Junction, Colorado. This proposal was approved and the necessary funds provided for construction of the plant.

Construction was started on November 1, 1952. The building was completed on January 19, 1953 and equipment was installed immediately thereafter. Resin-in-pulp equipment consisted essentially of six units or "banks" each containing four stainless steel screen "baskets" measuring 17" x 17" x 17". This equipment is described in detail in section IV. The pilot plant started on March 15, and by May 15, 1953, the process was operation satisfactorily from a metallurgical standpoint.

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2/ ACCO-3, 4, 6, 26, L 13
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From June 1, 1953, to September 1, 1953, 226.79 tons of copper uranium ore and 184.41 tons of Happy Jack ore, both from the White Canyon Area of Utah, were tested. These ores were dried and crushed to minus 10 mesh at the Monticello mill prior to shipping by truck to Grand Junction for treatment in the Pilot Plant. Rohm and Haas XE-123 plus 20 mesh resin was used.

II. OBJECT OF INVESTIGATION

The purpose of the Pilot Plant was to determine the commercial feasibility of the resin-in-pulp process, mechanically and metallurgically, as applied to uranium ores.

III. SUMMARY AND CONCLUSIONS

From six to 12 tons of ore per day were treated without difficulty. Desanding prior to resin-in-pulp was necessary for good mechanical operation. After the shakedown period, 99 per cent of the dissolved uranium was recovered from the pulp treated by the adsorption units. Overall pilot plant recoveries, based on head and tail assays and weight of feed, were about 83 per cent and 93 per cent respectively, for samples of copper uranium ores from Monticello stockpile 19 and from the Happy Jack Mine.

Mechanical and attrition losses of resin varied considerably as measured over two operation periods. From May 5, 1953, to July 1, 1953, resin loss amounted to 0.008 cubic feet per ton of ore treated or 0.003 cubic feet per pound of uranium recovered. A later check for the period from July 1, 1953, to August 9, 1953, showed that resin loss had dropped to 0.002 cubic feet per ton treated or 0.0004 cubic feet per pound of uranium produced.

The elutriant was 0.9 normal NH_4NO_3 solution acidified to a pH of one with sulfuric acid. The eluate was neutralized and yellow cake precipitated with anhydrous ammonia. Consumption of eluting and precipitation reagents varied widely and was substantially above that expected in normal operation. The test work necessitated frequent elution of partially loaded banks, and the subsequent precipitation of large volumes of low grade solutions consumed large quantities of NH_3. Loadings varied from a low of 1.5 pounds per cubic foot to a high of 3.7 pounds per cubic foot of wet settled resin, with an average of the order of 2.5 pounds per cubic foot.

Consumption of eluting and precipitation reagents for the month of August 1953 was as follows:

Table I

Consumption of Elution and Precipitation Reagents

Reagent	Lbs/Ton of Ore	Lbs/Lb of U_3O_8
NH_3	8.8	1.8
NH_4NO_3	18.3	3.83
H_2SO_4	29.98	5.35

Average consumption of leaching reagents is presented in Table II on the following page. No claim is made that these figures are optimum. Quantities used were based on pH measurements of leach solutions and information gained from bench scale tests prior to the pilot plant runs. No experiments were conducted to determine optimum leaching conditions.

Table II

Reagent Consumption in the Leaching Circuit

Ores	H_2SO_4 Lbs/Ton	MnO_2 Lbs/Ton
Stockpile 19	145	11.0
Happy Jack	115	18.4

Maintenance costs on the adsorption units from March 15 to August 31, 1953, were approximately $25.00.

Average grade of precipitate was only 40.3 per cent U_3O_8 but the final product was neither washed nor calcined during the test period. Laboratory tests and subsequent pilot plant work has indicated that washing and calcining will bring the grade to about 70 per cent U_3O_8. The contaminant are, for the most part, iron, caused by incomplete loading, soluble salts from liquors retained in yellow cake, and acid insoluble material.

The pilot plant work on the White Canyon ores has demonstrated that the resin-in-pulp process is entirely feasible, even though proper equipment for producing high grade product at lowest cost was not available. We conclude that the process should definitely be considered where settling problems are anticipated and it may, with further development, be competitive with CCD and columns where both methods are applicable.

IV. MECHANICAL DEVELOPMENT

A. Investigation of Proposed Methods

In the early resin-in-pulp investigations at the laboratory, then located at Watertown, Mass., many suggestions and proposals were advanced involving various gadgets and machines all of which could be classified into one or the other of two basic procedures as follows:

1. A system in which the resin actually moves countercurrent to the flow of pulp in the loading cycle and countercurrent to the flow of eluant in the eluting cycle.

2. A modified countercurrent system in which the resin is confined to containers or units through which the pulp or eluant flows, the countercurrent effect being produced by intermittently advancing each unit in its respective circuit.

The procedure typical of (1) above, under consideration at that time, was exemplified by a system of trommels and agitators in which the beads were advanced from tank to tank by elevating with lifter bars. This system had the apparent advantage of positive countercurrent effects, low capital cost, and ease of operation. Its possible disadvantages were high resin loss and low screening capacity.

The basket machines installed in the pilot plant were adaptable to the modified countercurrent system. This system evolved from early bench scale tests in which "tea bags" or small stainless steel baskets were used to contain the resins. Figure 1, on pages 11 and 12, is a flowsheet showing the system as used in the pilot plant.

B. Construction

Each basket was made up of a hard maple framework rigidly supported and held together by tongue and groove joints. The top of the basket was left open with the remaining five sides enclosed with Ludlow Saylor #281 Rek-Tang Screen bolted to the framework with 1/4" x 2 1/4" machine bolts. The bolts were stainless steel while the screen was woven with Carpenter 20.

Two baskets were alternately raised and lowered 16 times per minute through a four inch stroke by means of an eccentric actuated by a one-fourth horsepower gearmotor with chain and sprocket drive. A cross section of this mechanism and basket appears in the sketch in Figure 2, page 12. Six units or "banks" were constructed of redwood with tongue and groove joints. Each bank was divided into four "cells" to accommodate four baskets. At the discharge end of each bank, a second partition was installed to permit overflow into an airlift. Another inlet was also provided inside of the machine; when this inlet was opened the airlift would elevate all of the pulp from the machine. This inlet was opened to drain the machine between exhaustion and elution cycles. This arrangement is pictorially sketched in Figures 3 and 4 on pages 14 and 15, respectively. The satisfactory manner in which the airlifts functioned had much to do with the successful operation of the units. The discharge of the airlift can be directed either to precipitation, the next succeeding bank, or to tailings. All of the component parts of the adsorption units were fabricated locally.

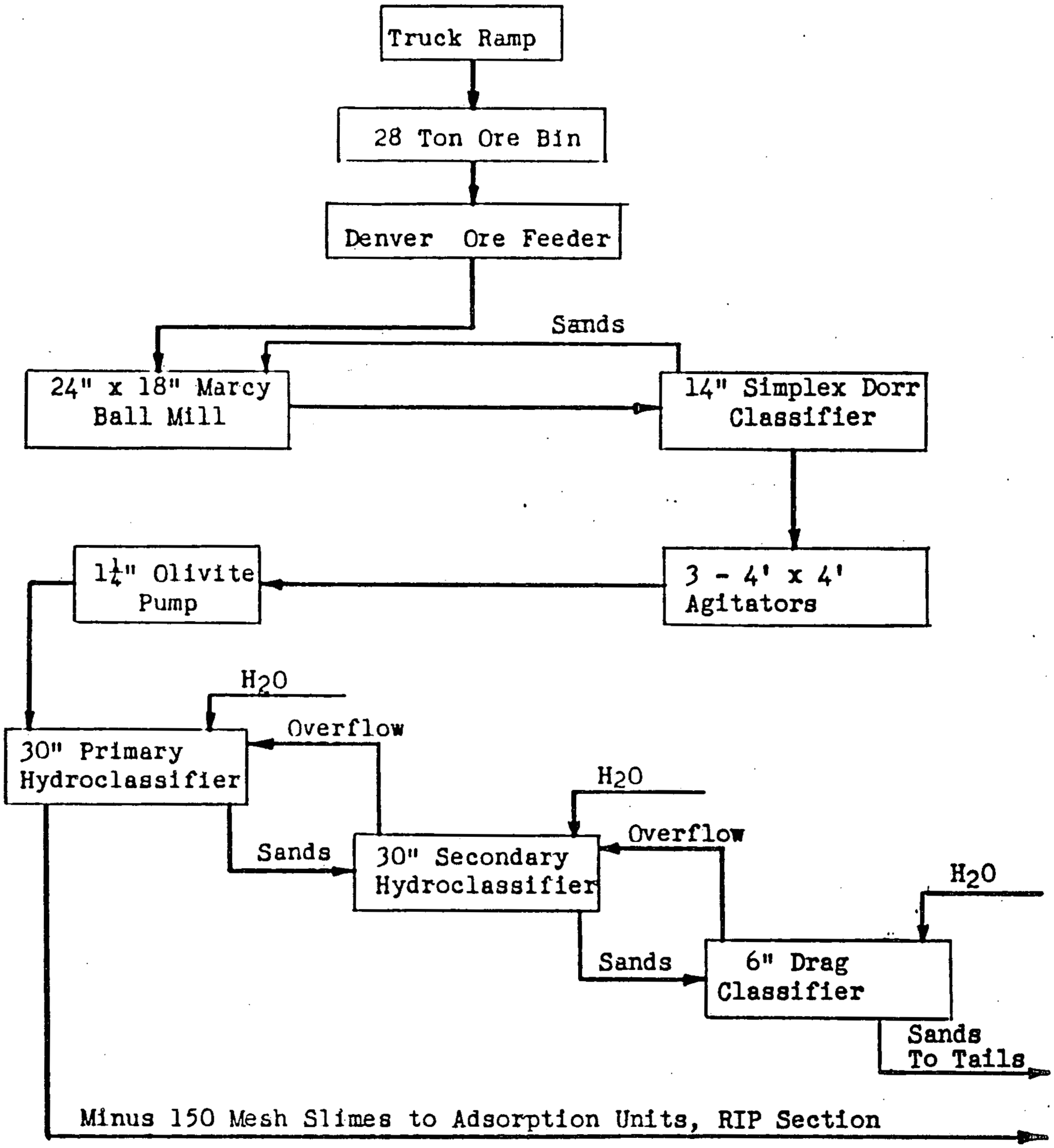

Figure 1. Pilot Plant Flowsheet
Leaching and Classification Section

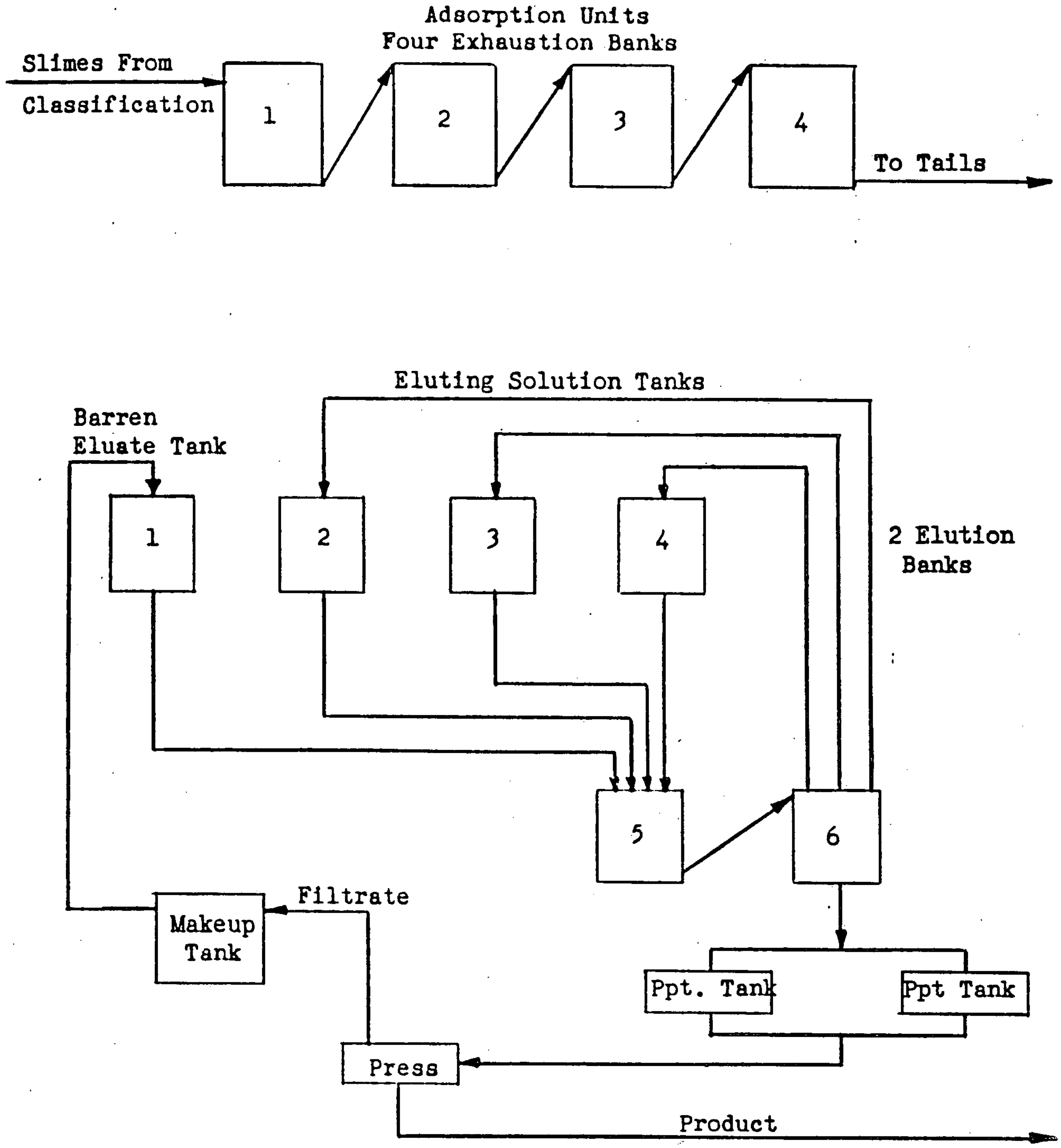

Figure 1 (Continued). Pilot Plant Flowsheet
Resin-in-Pulp Section

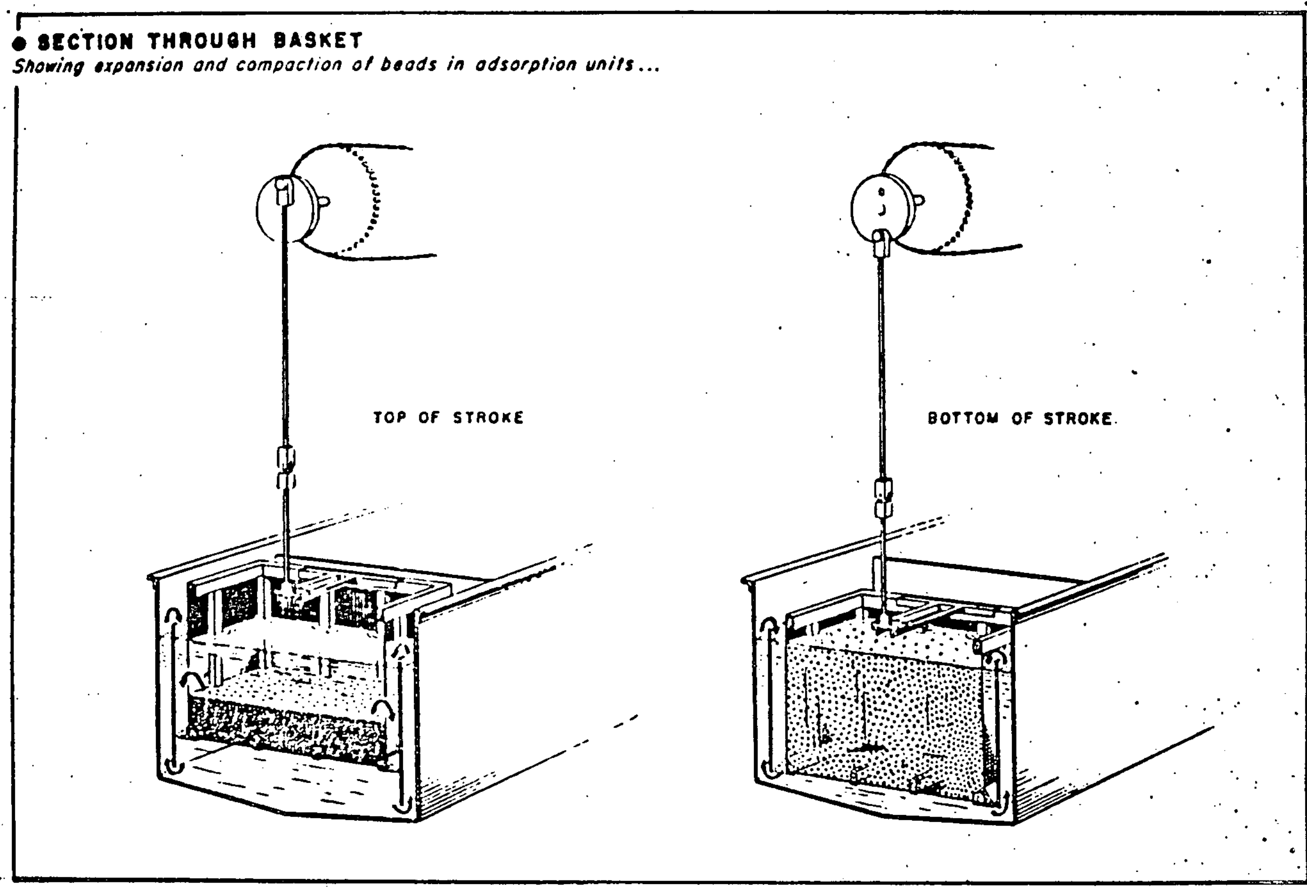

Figure 2. Section Through Basket
(Showing Expansion and Compaction of Beads in Adsorption Units)

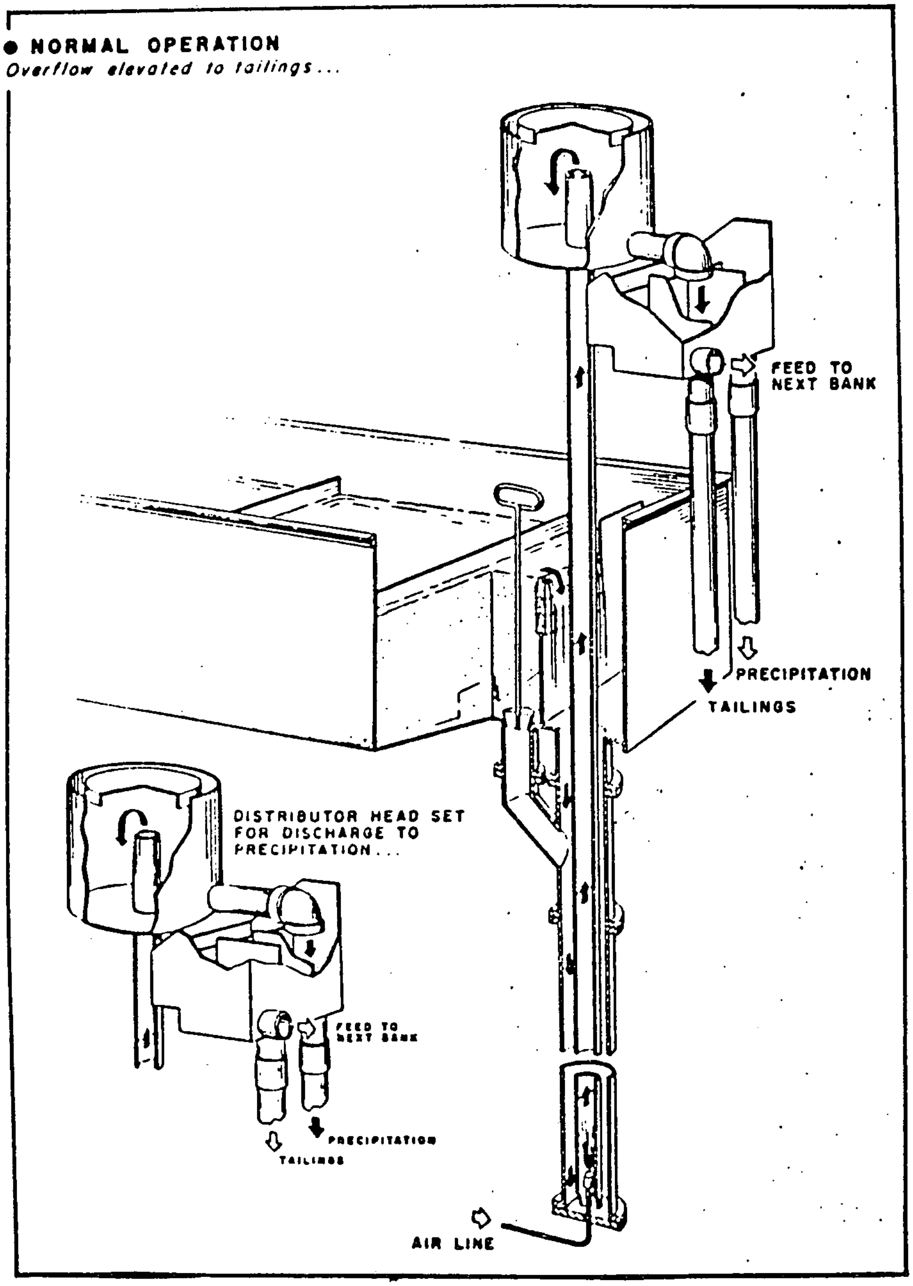

Figure 3. Normal Operation
(Overflow Elevated to Tailings)

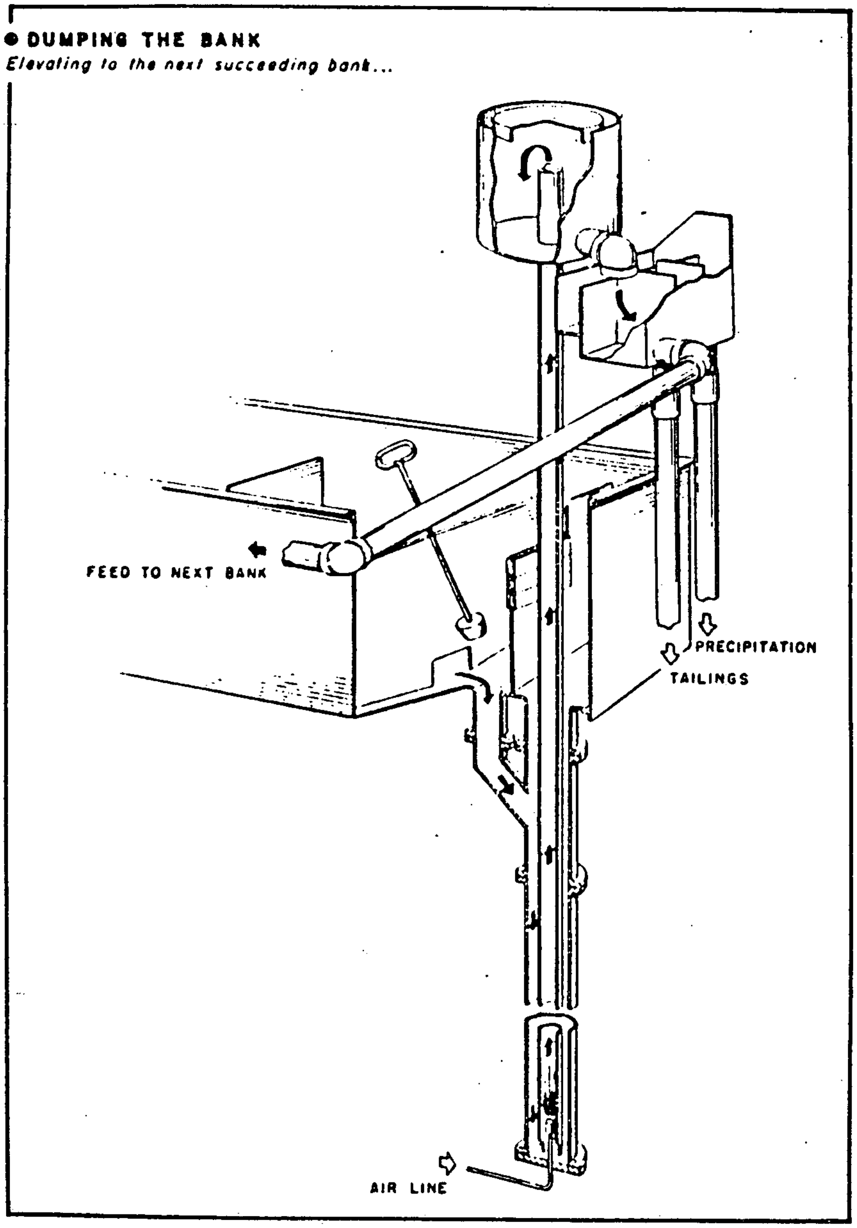

Figure 4. Dumping the Bank
(Elevating to the Next Succeeding Bank)

V. TEST WORK PROCEDURES

A. Crushing

All ore tested in the pilot plant was crushed to minus 10 mesh at the Monticello mill and hauled by truck to Grand Junction as needed.

A typical screen analysis of a crushed sample of copper uranium ore from stockpile 19 is shown in Table III, below.

Table III

Screen Analysis Monticello Crusher Product

Tyler Mesh		% Weight	Cum. % Weight
+10		7.83	7.83
-10+20		19.11	26.94
-20+28		10.18	37.12
-28+35		14.06	51.18
-35+48		16.97	68.15
-48+65		7.66	75.81
-65+100		6.80	82.61
-100+150		5.50	88.11
-150+200		3.66	91.77
-200		8.23	100.00
	Total	100.00	

B. Grinding

The crushed ore, on arrival in Grand Junction, was weighed and then dumped into a 28-ton ore bin. From the mill bin the ore was fed at a rate of 500 to 600 pounds per hour to a 24" x 18" Marcy ball mill in closed circuit with a 14 inch Dorr classifier. The classifier overflow was regulated in a range from 50 to 60 per cent solids. A representative screen analysis of the classifier overflow on a sample of copper uranium ore appears in Table IV, below.

Table IV

Screen Analysis Classifier O'flow 50 to 55 Per Cent Solids

Tyler Mesh	% Weight	Cum % Weight	% U_3O_8	Cum % U_3O_8
+20	Trace			
-20+28	0.93	0.93	0.18	0.18
-28+35	3.94	4.87	0.14	0.15
-35+48	11.28	16.15	0.12	0.13
-48+65	25.16	41.31	0.14	0.14
-65+100	5.42	46.73	0.11	0.14
-100+150	11.18	57.91	0.13	0.14
-150+200	5.93	63.84	0.15	0.14
-200	36.16	100.00	0.27	0.17
TOTAL	100.00		0.168	

C. Leaching

Classifier overflow was elevated by a one inch Wilfley pump to three, four by four foot Devereaux Type agitators which served a leaching tanks. Contact time when feeding 500 pounds per hour at 50 per cent solids was, theoretically, 10 hours. Acid addition was made to the No. 1 agitator and was controlled by maintaining the pH in No. 3 agitator at approximately 1.0.

Average acid consumption by ore from Stockpile 19 was 145 pounds per ton of ore. Happy Jack acid consumption was of the order of 115 pounds per ton. In addition, 11.0 pounds of MnO_2 per ton of feed was used on Stockpile 19 ores while 18.4 pounds of MnO_2 per ton of ore was used when treating material from the Happy Jack Mine. No attempt was made to determine whether or not these reagent quantities were optimum. The additions were governed somewhat by bench scale tests conducted in the Winchester Laboratory.

D. Sand-Slime Separation

In the early test work at Watertown it was apparent that a desanded pulp could be handled much easier in almost any device designed for R.I.P. processes. A short test run in the pilot plant without a sand-slime separation prior to treatment in the adsorption units confirmed this early conclusion. Sand collected in the baskets to such an extent that satisfactory operation was impossible. Thereafter, all pulp fed to the adsorption units was desanded in two 30" hydroclassifiers and one small drag classifier. Wash water and overflows moved countercurrently with respect to the sands which reduced soluble uranium losses to about 2.5 per cent of that in the leach liquor.

E. Adsorption

Adsorption of uranium from the diluted and desanded pulp was effected in a series of machines, each of which had four baskets 17" x 17" x 17" filled with 0.68 cubic feet of wet settled resin. Each basket moved up and down in the pulp 16 times a minute. Operation of the units was controlled by periodically assaying the soluble uranium in the pulp as it flowed from the last machine to tails. When the uranium in the liquor reached a certain level, usually two per cent of that in the feed, the first bank on the loading cycle was taken out and a freshly eluted bank was added to the circuit.

F. Elution

As each loaded bank was taken off the adsorption circuit, the pulp was drained and the machine washed with a water spray to remove residual solid material. Elution was then effected by advancing six 200 gallon batches of eluting solution. The first batch was advanced and sent to the precipitation tanks and each succeeding batch went to the machine then advanced to the next eluant tank. These batches in turn were advanced on the succeeding cycles.

G. Precipitation and Filtration

Eluate from the first pass was dumped into a five by five foot stainless steel mixing tank and neutralized with anhydrous ammonia to precipitate the uranium. The slurry was pumped through a 12 plate 24" x 24" Shriver Press.

H. Eluting Solution Makeup

Filtrate from the press flowed by gravity to a storage tank from which point it was pumped as needed to a mixing tank where sufficient ammonium nitrate was added to bring the nitrate strength back to 0.9 normal. Sulfuric acid was then added until the solution had a pH of 1.0. This regenerated solution was then pumped as needed to the first (Barren) eluting solution tank.

VI. ORE TESTING

A. Preliminary Operation

On completion of the mill in late March 1953, a test run was started using Temple Mountain ore after first having run barren bentonitic material which served to demonstrate mechanical weakness without sacrificing uranium. In the trial run on barren material no acid was used to simulate leaching. Following a siege of leaky tanks, Temple Mountain ore was then leached and pumped directly to the units without a sand-slime separation prior to treatment in the adsorption units.

Within a week it was apparent that operation without first removing the coarse sands was impractical. The baskets plugged repeatedly and operation in general was completely unsatisfactory. Thereafter, a sand-slime separation was made with only the minus 150 mesh material going to the adsorption units and operation from then on was mechanically trouble free.

Metallurgically the results, during the first three weeks, were disappointing. Recovery of uranium from leach liquors was of the order of 96 per cent where 99+ per cent was expected. As time went on, recovery steadily dropped until it became obvious that either the resin beads were poisoned or Temple Mountain ores were not amenable to ion exchange processes. Unfortunately, no bench scale column tests had been run on the ore prior to treatment in the plant.

Test work on Temple Mountain ore was discontinued and a run was started on material from Stockpile 19, a copper uranium ore from the White Canyon district. Metallurgically the performance of the units continued discouraging with tails steadily rising. The beads were then eluted with a one normal solution of HNO_3 without any appreciable benefit. Ultimately, as tailing losses became greater, the resin was treated with a four normal HNO_3 solution. This apparently eluted the deleterious material from the resin for the adsorption of dissolved uranium immediately became 99 per cent or greater.

Later investigation failed to disclose the cause of the apparent resin poisoning.

B. Leaching & Adsorption - Stockpile 19 - Copper Uranium Ore from White Canyon Area of Utah

After the early metallurgical difficulties were overcome, adsorption from the leach liquors was satisfactory, when treating ore from Stockpile 19. Leaching in the plant confirmed bench scale tests. No trouble was experienced in running at the rate of six tons per day and runs up to 1000 pounds per hour were tried with little change in metallurgical results. The ability to run with high feed rates, however, must be attributed to the relatively low grade feed which averaged only 0.155 per cent U_3O_8.

From June 1 through July 18, 1953, 226.79 tons of ore were treated in the plant with metallurgical results as outlined in Table V.

Table V

Metallurgical Data
White Canyon Copper Uranium Ore

June 1 - July 18

	Tons Treated	% U_3O_8	Content Lbs U_3O_8	% Total
Feed	226.79	.155	703.05	100.00
Residue	215.45	.022	94.80	13.48
Leach Liquor			608.25	86.52
Soluble Loss Class Sands			17.96	2.56
Adsorption Unit Feed (Hydro-Class O'flow)		.50 g/L	590.29	83.96
Yellow Cake (By Difference)			584.45	83.13
Adsorption Unit Tails (Solutions Only)		.005 g/L	5.84	0.83

Adsorption unit recovery = 99.00%
Overall plant recovery = 83.13%*

* Does not take into account tank and pump leakage or losses in water washes which would be recycled in commercial operation.

Operating conditions - Feed rate 500 - 1000 lbs/hr.
Acid consumption - 145 lbs/ton
Oxidant - MnO_2 - 11.0 lbs/ton

During the test run on Stockpile 19, two methods of loading and eluting were tried. When the feed rate was set at 500 pounds per hour it was possible to run with five banks on exhaustion and one on elution. This permitted a changeover every six to eight hours. With higher feed rates "breakthrough" would occur before the elution cycle was complete, therefore, it was necessary to load on four banks and elute two banks at one time. Elution was performed by pumping through four batches of 350 gallons of eluant which had been advanced through preceding elution cycles. Only one batch of 350 gallons was precipitated each cycle which gave a volume ratio of final eluate to wet settled resin of 8.1:1.

C. Leach and Adsorption - Happy Jack Ore

On July 21, 1953, a run was started on ore from the Happy Jack mine. This material was higher grade than that from Stockpile 19. Therefore, feed rates varied from 500 to a maximum of 750 pounds per hour. In general, extraction was above 96.8 per cent and recoveries from the resultant leach liquors were very high. Loadings reached a maximum of 3.7 pounds per cubic foot and averaged approximately 2.75 pounds per cubic foot.

Generally speaking, Happy Jack ore was very amenable to the process both mechanically and metallurgically.

From July 21, 1953 to August 31, 1953, 184.81 tons were treated in the plant with recovery as indicated in Table VI.

Table VI

Metallurgical Data
Happy Jack Ore

July 21 - August 31

	Tons Treated	% U_3O_8	Content Lbs U_3O_8	% Total
Feed	184.81	.300	1108.86	100.00
Residue	175.57	.011	38.63	3.48
Leach Liquor			1070.23	96.52
Soluble Loss Class Sands			32.11	2.90
Adsorption Unit Feed (Hydro-Class O'flow)		1.07 g/L	1038.12	93.62
Yellow Cake (By Difference			1030.33	92.92
Adsorption Unit Tails (Solutions Only)		.008 g/L	7.79	0.70

Adsorption unit recovery = 99.25%
Overall plant recovery = 92.92%*

*Does not take into account tank and pump leakage or losses in water washes which would be recycled in a commercial operation.

Operating conditions - Feed rate 500 - 700 lbs/hr.
Acid consumption - 115 lbs/ton
Oxidant - MnO_2 - 18.4 lbs/ton

D. Elution and Precipitation

Elution in all of the test work was performed by passing successive batches of eluant through the banks of loaded resin. A "bump" was always experienced in the barren assay after elution but this was never serious as long as the last eluant pass was below .25 grams per liter. Eluant was advanced from cycle to cycle so that a countercurrent effect was reached permitting precipitation of a solution as high as 7.0 grams U_3O_8 per liter. The eluant was 0.9 normal ammonium nitrate solution acidified to a pH of 1.0 with sulfuric acid. In continuous operation eluant was made up from the recycled press filtrate.

Precipitation was accomplished by neutralization with anhydrous NH_3 to a pH of seven.

E. Precipitate Grade

At the end of the exhaustion cycle the slime in the bank was dumped through the succeeding banks. Then the loaded bank was filled with water and the wash subsequently dumped to tails. Residual solids were partially sluiced out with a hose. When eluting solution was pumped into the banks some solids were picked up and sent to precipitation and ultimately showed up in the yellow cake. This resulted in a somewhat lower grade product than experienced in column work.

Several shipments of yellow cake were made to the A.S. & R. sampling plant during the period from May 5, 1953 to August 31, 1953. These shipments averaged 40.3 per cent U_3O_8.

Laboratory tests were run on several barrels of final product to determine the effects of washing and calcining. The results are outlined in Table VII.

Table VII

Effects of Washing and Calcining on Grade of Precipitate

Barrel No.	Untreated % U_3O_8	Washed % U_3O_8	Calcined % U_3O_8
1	48.2	73.1	80.4
2	35.5	--	50.0
3	37.3	--	62.5
4	38.1	75.9	85.9
5	20.7	38.2	--
6	30.2	62.1	68.9

In general, washing and calcining almost doubled the grade of the yellow cake. Four grab samples of product assayed 6.53 per cent, 11.39 per cent, 9.80 per cent and 16.00 per cent insoluble.

The results of the tests indicated the principal contaminants in the yellow cake were: (1) Iron caused by incomplete loading, (2) Soluble salts from eluate retained in the yellow cake, (3) Acid insoluble material from incomplete washing in the banks prior to elution.

F. Resin Losses

In the early shakedown period of operations accurate measurement of bead losses was impossible because of spills, removal of resin for special test work, and losses from inspection of baskets. However, when operation had settled down to a regular routine, 19.54 liters of wet settled resin were accurately measured and added to each basket. In the period from May 5, 1953 to July 1, 1953, 0.008 cubic feet of wet settled resin was lost per ton of ore treated. This represented, on the basis of pilot plant operation, 122.6 per cent loss per year. This loss was considered abnormally high because some spills had occured in the period. This supposition was borne out by the losses as measured in the next period from July 1 to August 9, 1953. In this period 0.002 cubic feet of wet settled resin was lost per ton of ore treated. This represented a loss of 29.7 per cent in a one year operation.

Re-adjusting the data above in terms of U_3O_8 produced, the first period resulted in a loss of .003 cubic feet of wet settled resin per pound of U_3O_8 adsorbed, whereas, in the second period, this loss dropped to 0.0004 cubic feet of wet settled resin per pound of U_3O_8 adsorbed.

Generally speaking, the mechanical performance of the units and the metallurgical results obtained, were most encouraging. New operating techniques have continually developed since the preliminary test runs and these techniques have further improved operating efficiencies and should reflect in even better performance in the future.